

Original Research Article

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## Treatment of Electroplating Cr(VI) for Reduction Cr(VI) by Electrocoagulation in Continuous Operation

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### ABSTRACT

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The performance of electrocoagulation, with aluminium sacrificial anode, in the treatment of metal ion Cr(VI) containing wastewater, has been investigated. Several working parameters, such as pH, current density and metal ion concentrations were studied in an attempt to achieve a higher removal capacity. Results obtained with synthetic wastewater revealed that the most effective removal capacities of studied metal could be achieved when the pH was kept between 4 and 8. In addition, the increase of current density, in the range 0.8–4.8Adm<sup>-2</sup>, enhanced the treatment rate without affecting the charge loading, required to reduce metal ion concentrations under the admissible legal levels. The process was successfully applied to the treatment of an electroplating wastewater where an effective reduction of Cr(VI) concentrations under legal limits was obtained, just after 20 min. The electrode and electricity consumptions were found to be 1 g l<sup>-1</sup> and 32 A h l<sup>-1</sup>, respectively. The method was found to be highly efficient and relatively fast compared to conventional existing techniques.

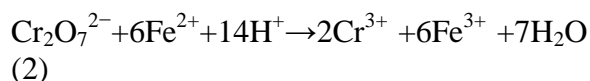
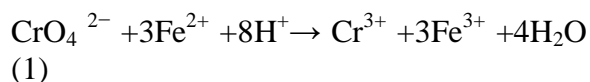
## Introduction

Water pollution by heavy metals, especially chromium; have sparked much concern to societies and regulation authorities around the world. Due to chromium wide usage by different industries such as metal plating, paints and pigments, leather tanning, textile dyeing, printing inks and wood preservation, huge quantity of wastewater containing chromium is discharged into the environment in trivalent (Cr(III)) and hexavalent (Cr(VI)) forms. The hexavalent chromium compounds are toxic and carcinogenic. In contrast, relative toxicity of Cr(III) is low and in its trace amounts, it is not a problem for the environment [Fu, 2011].

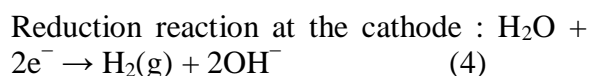
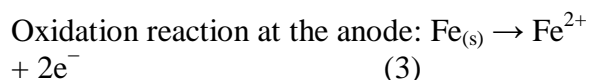
The most common method used for the removal of chromium from wastewater is the acidic reduction of Cr(VI) to Cr(III) (pH 2–3) followed by raising pH to precipitate the Cr(III) [Jin *et al.*, 2010]. Therefore, chromium reduction is an important phenomenon since it converts toxic mobile Cr(VI) into less toxic immobile Cr(III) [Zhitkovich, 2011]. Cr(VI) can be removed from aqueous waste by a variety of techniques, such as chemical reduction followed by precipitation, ion exchange, reverse osmosis and adsorption. The conventional treatment application currently used to remove Cr(VI), is its reduction to Cr(III) by chemical means followed by precipitation of Cr(OH)<sub>3</sub> [CaO and Jin, 2015; Jin *et al.*, 2011; Pereira *et al.*, 2012; Wei *et al.*, 2015]

In order to remove Cr(VI) by chemical means, it is necessary to perform reduction of Cr(VI) to Cr(III) at acidic pH (2.0–4.0) and precipitation of formed Cr(III) at alkaline pH (7.0–10.0) conditions, in a two-step process. Cr(VI), which can be found in aqueous waste as either chromate (CrO<sub>4</sub><sup>2-</sup>),

or dichromate (Cr<sub>2</sub>O<sub>7</sub><sup>2-</sup>) species, can be reduced to Cr(III) by dissolved Fe(II) according to equations given below:



As is evident from Eqs. (1) and (2), reduction from Cr(VI) to Cr(III) requires acidic media and a Fe(II) source in order to shift the equilibrium to the right-hand side; therefore, continuous proton and Fe(II) sources have to be supplied. In the electrocoagulation (EC) process both reduction and precipitation take place in the same reactor. The electrochemical process involves the liberation of ions into the solution due to the anodic polarization of electrode. In an EC cell the following simultaneous reactions occur when aluminum electrodes are employed:



During these reactions, the Fe(II) ions released at the anode causes reduction of Cr(VI) species to Cr(III). Oxidized aluminum (Fe(III)) combines with the hydroxyl ions produced at the cathode to form the precursor of the insoluble ferric hydroxide (Fe(OH)<sub>3</sub>) or goethite (FeOOH) matrix, necessary for the precipitation of Cr(III) species [Malaviya and Singh, 2011]. Cr(III) may be removed through the precipitation of Cr(OH)<sub>3</sub>, adsorption onto goethite or substitution with Fe(III) in the Fe(OH)<sub>3</sub> [Malaviya and Singh, 2011; Miretzky and Cirelli, 2010]. A great deal of work performed in the last decades has

proven that electrochemical treatment using aluminum and aluminum electrodes is an effective method for the reduction of Cr(VI) [Pereira *et al.*, 2012; Malaviya and Singh, 2011; Jin *et al.*, 2013; Jin *et al.*, 2014]. However, these studies have mostly been conducted on synthetic solutions containing low concentrations of Cr(VI) [Pereira *et al.*, 2012; Ioannou *et al.*, 2015; Hasin *et al.*, 2010; Jin *et al.*, 2012]. Dissolved oxygen may interfere with the reaction between Cr(VI) and Fe(II) by its own ability to oxidize Fe(II). It is well known that Fe(II) oxidation by dissolved oxygen is primarily dependent on the pH and the dissolved oxygen concentration of an aqueous solution [Arar *et al.*, 2013]. Schlautman and Han [Dubrawski *et al.*, 2014] concluded that the effect of dissolved oxygen on Fe(II)–Cr(VI) reaction will be minor, particularly for lower pH values. Previous researchers also stated that the presence of dissolved oxygen is expected to be important only at pH values greater than 8.0 [Dubrawski *et al.*, 2014; Meas *et al.*, 2010].

In the present work, the efficiency of electrocoagulation in removing chromium (Cr(VI)) from wastewater of an electroplating unit was reported. The effect of the wastewater characteristics, initial pH and metal-ion concentrations and operational variables, current density and treatment time, on the removal efficiency is explored and discussed to determine the optimum operational conditions. The optimum operational parameters were used for wastewater treatment of a local electroplating unit.

## **Materials and Methods**

### **Materials and Instruments**

The Cr(VI) solution was prepared by dissolving potassium dichromate (Merck,

Germany) in distilled water. The conductivity of solutions was raised up and adjusted by adding one of NaCl, Na<sub>2</sub>SO<sub>4</sub> or NaNO<sub>3</sub> salts as electrolyte (Fluka, Switzerland). The conductivity measurement was carried out using a HACK conductivity meter. The pH of the solutions was measured by pH meter (Metrohm 654, Switzerland) and adjusted by adding NaOH or HCl (Merck, Germany) solutions. aluminum (HE 18) plates were used. Dimensions of electrodes were 150mm\*160mm\* 1mm. The electrodes were connected to a DC power supply (ADAK PS808, Iran) with current static operational option to control the current density.

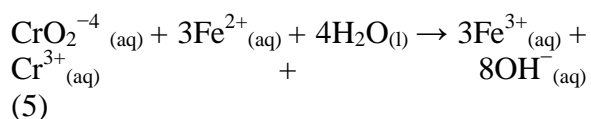
### **Electrocoagulation Test**

A laboratory-scale reactor (24 cm×17 cm×18 cm), made of Plexiglas sheets, was used. There were 4 metal plates (electrode), each dimension was 150mm\*160mm\* 1mm, from Aluminum in the tank. Plates were installed in parallel configuration with 1.5cm interspaces. A magnet was placed in the bottom of the tank for mixing (was adjusted in 300 rpm). Settling chamber was made from Plexiglas in 24(depth)\*17(width)\*53(length) and 21.5l in volume (for 30minutes contact time) and electrocoagulation and settling reactor were attached to each other in series. After the considered contact time, solution enters to settling chamber from electrocoagulation tank. In settling chamber 3 valves were implanted for providing the 30 min contact time which their location was determined previously. Solution passes slowly through the settling chamber and finally, the treated water comes out from valve. Samples were collected from tap when the water level in tank reached to the mean of the sampling tap. Four 12\*15cm metal plates (electrodes) from wrought (HE 18) Aluminum with 1mm diameter were used for this study.

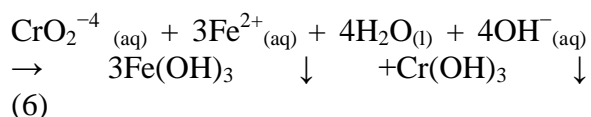
Electrodes were placed parallel with 1.5 cm interspace. Before each test, electrodes were rubbed by sandpaper and washed by sulfuric acid 1N. To supply DC current with adjustable amperage (0-60 V, 0-6 A), a transformer was used. Since electrode surface and electrode interspace are constant, by dissolving sodium chloride (increasing specific electrical conductivity of solution) electric current intensity was adjusted. Current level was adjusted for 0.8, 1.6 and 3.2 A. So, by dividing current level to total surface of anode, current density is obtained that is 0.12, 0.24, 0.48 ampere per m<sup>2</sup>, respectively.

### Brief Description of Electrocoagulation Mechanism

Wastewater containing Cr<sup>6+</sup> (CrO<sub>4</sub><sup>2-</sup>) ions can be removed by the EC technique using aluminum as the sacrificial anode [19]. The ferrous ion (Fe<sup>2+</sup>) or Al<sup>2+</sup> generated by electrooxidation of the aluminum anode can reduce Cr<sup>6+</sup> to Cr<sup>3+</sup> under alkaline conditions and is itself oxidized to ferric (Fe<sup>3+</sup>) or Al<sup>3+</sup> ion according to

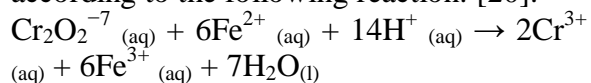


Or



The Cr<sup>3+</sup>(aq) ion is then precipitated as Cr(OH)<sub>3</sub>(s) by raising the pH of the solution. The Fe<sup>2+</sup>(aq) ions can also reduce

Cr<sub>2</sub>O<sub>7</sub><sup>2-</sup>(aq) under acidic conditions according to the following reaction: [20].



### Results and Discussion

The electrocoagulation process is quite complex and may be affected by several operating parameters, such as pollutants concentrations, initial pH and current density. In order to enhance the process performance, the effects of those parameters have been explored.

It has been established that the initial pH [Barrera-Diaz *et al.*, 2011; Barrera-Diaz *et al.*, 2014] has a considerable influence on the performance of electrocoagulation process. To evaluate its effect, a series of experiments were performed, using solutions containing of Cr(VI) 50 mg l<sup>-1</sup>, with an initial pH varying in the range 2–10. As illustrated in Fig. 1, the removal efficiencies (*Re*) of Cr(VI) when pH exceed 4. In the 15 min of electrolysis at 0.8 A conditions, the removal yield of chromium reached a maximum of about 83% and seemed to be not affected by pH, as long as this later is kept in the range between 4 and 8. In contrast, when the initial pH is increased above 8, a dramatic decrease of the removal efficiency of chromium is observed (58%), while removal yield of Cu<sup>2+</sup> remained very high. Furthermore, it can be seen that the removal efficiency of all studied ions decreased significantly upon decreasing initial pH. Removal yield lower than 55% was achieved at pH 2. The decrease of *Re* at a pH less than 4 and higher than 8 was observed by many investigators and was attributed to an amphoteric behavior of Al(OH)<sub>3</sub> which lead to soluble Al<sup>3+</sup> cations, when the initial pH is low and to monomeric anions Al(OH)<sub>4</sub><sup>-</sup>, when the initial pH is high. These soluble species are useless for water treatment. When the initial pH was kept in the range 4–8, all aluminum cations produced at the anode formed polymeric species Al<sub>13</sub>O<sub>4</sub>(OH)<sub>24</sub><sup>7+</sup> [20,22] and precipitated Al(OH)<sub>3</sub> leading to a more effective treatment. At alkaline pH between

8 and 10, dichromate ions are converted to soluble chromate ( $\text{CrO}_4^{2-}$ ) anions, which goes some way towards explaining its less effective removal.

As observed by other investigators [Tian *et al.*, 2012; Yang *et al.*, 2014], the treatment induced an increase in the pH when the initial pH was low (between 2 and 9) as shown in Fig. 2. This might be explained by the excess of hydroxyl ions produced at the cathode in sufficiently acidic conditions and by the liberation of  $\text{OH}^-$  due to the occurrence of a partial exchange of  $\text{Cl}^-$  with  $\text{OH}^-$  in  $\text{Al}(\text{OH})_3$ . When the initial pH is above 9, the formation of  $\text{Al}(\text{OH})_4^-$  species together with parasite attack of the cathode by hydroxyl ions [He and Angenent, 2009] lead to a slight decrease in the pH. As a result of the previous discussion of the effect of pH on the removal efficiency, the initial pH was adjusted to 6 for all subsequent studies.

### The Effect of Current Density

The current density not only determines the coagulant dosage rate, but also the bubble production rate and size [Huang *et al.*, 2011; Liu *et al.*, 2011]. Thus, this parameter should have a significant impact on pollutants removal efficiencies. To investigate the effect of current density and charge loading on the removal yield, a series of experiments were carried out on solutions containing a constant pollutants loading with current density being varied from 0.8 to  $4.8 \text{Adm}^{-2}$ . Fig. 3 is a semi logarithmic plot, showing the normalized concentrations profiles of the studied metal ions for typical electrocoagulation runs, where the initial pH was fixed at 6. The removal rate of all studied metal ions increased upon increasing current density. The highest current ( $4.8 \text{Adm}^{-2}$ ) produced the quickest removal rate, with a 96% concentration reduction occurring just after 10 min.

This expected behaviour is easily explained by the increase of coagulant and bubbles generation rate, resulting in a more efficient and faster removal, when the current is increased [Liu *et al.*, 2011; McCarty *et al.*, 2011]. Indeed, the amounts of aluminium and hydroxide ions generated at a given time, within the electrocoagulation cell are related to the current flow, using Faraday's law:

$$m = \frac{ItM}{zF} \quad (8)$$

where  $I$  is the current intensity,  $t$  is the time,  $M$  is the molecular weight of aluminum or hydroxide ion ( $\text{g mol}^{-1}$ ),  $z$  is the number of electrons transferred in the reaction (3 for aluminum and 1 for hydroxide) and  $F$  is the Faraday's constant ( $96486 \text{Cmol}^{-1}$ ). Moreover, it was previously shown that the bubble size decreases with increasing current density [An and Zhao, 2011], which is beneficial to the separation process. Nevertheless, as the time progresses, the amount of oxidized aluminum and the required charge loading increase. However, these parameters should be kept at low level to achieve a low-cost treatment. To optimize the treatment efficiency, optimum charge loading required to achieve high removal yields (residual concentration under  $2 \text{mg l}^{-1}$ ) for each metal ion, were calculated at different current densities. The results shown in Fig. 4, pointed out that the removal efficiency of chromium was  $59.4 \text{mFl}^{-1}$ . Furthermore, as observed by other investigators [Zhang *et al.*, 2013; Alvarado and Chen, 2014], a slight increase of charge loading is observed for chromium, when current density was varied in the range 0.8– $4.8 \text{Adm}^{-2}$ . At high current, the bubble density and upwards flux increased and resulted in a faster removal of the coagulant by flotation. Hence, there is a reduction in the probability of collision between the coagulant and pollutants. Indeed, Regardless



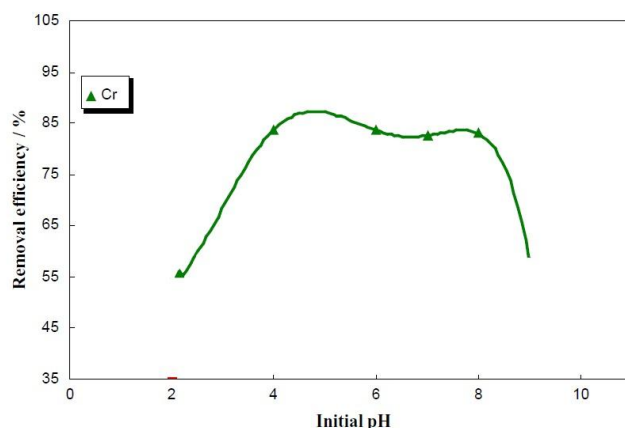
the slight increase (below 20%) of the charge loading observed for Cr(VI), the time required to achieve the treatment can be shortened by a factor of six, when current density is increased from 0.8 to 4.8  $\text{Adm}^{-2}$ , while the cost of the treatment remained unchanged. Hence, the highest current should be selected to obtain the quickest removal rate.

### The Effect of Metal Ion Concentration

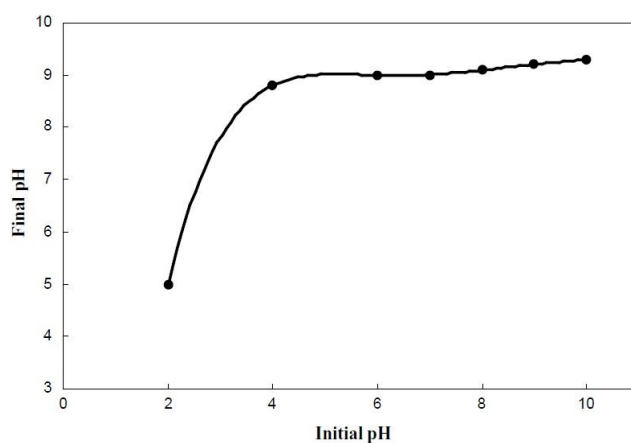
In order to examine the effect of metal ion concentration present in the wastewater on the removal rate, several solutions

containing increased concentrations (50–800  $\text{mg l}^{-1}$ ) of Cr(VI) were treated and the residual concentrations of ions were measured at different times of electrolysis. Fig. 3 shows the change in the removal rate of chromium removal rate. As expected, it appears that the removal rate has decreased upon increasing initial concentration. This induced a significant increase of charge loading required to reach residual metal concentrations below the levels admissible for effluents discharged into the sewage system (2  $\text{mg l}^{-1}$ ), as shown in Fig. 4. It can be observed that charge loading undergo a linear increase with initial concentration.

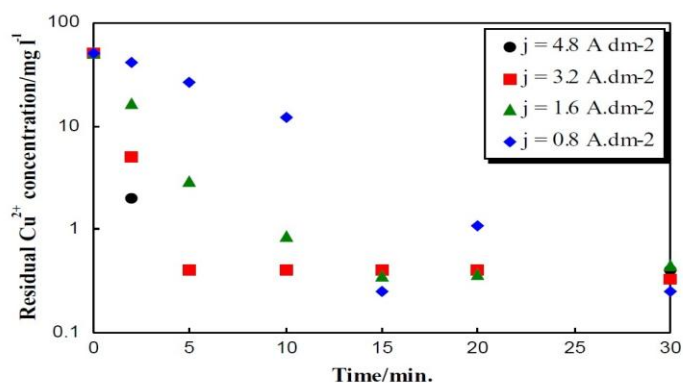
**Fig.1** Effect of Initial pH on Metal Ion Removal. Initial Concentrations of Cr(VI) = 50  $\text{mg l}^{-1}$  each,  $j = 1.6 \text{Adm}^{-2}$ , Anode Surface = 50  $\text{cm}^2$ , Time of Electrolysis = 20



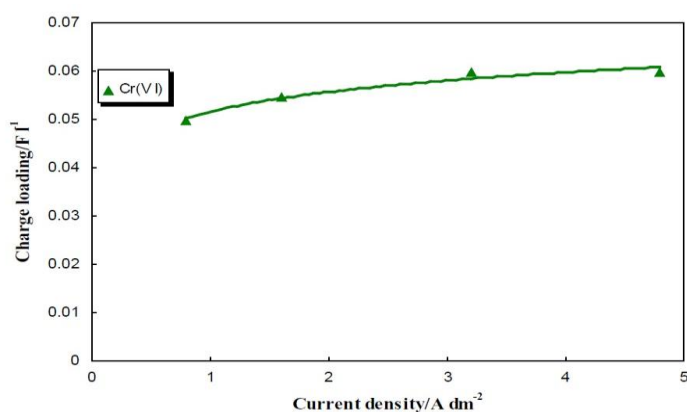
**Fig.2** pH Variation after Electrocoagulation. Initial Concentrations of Cr(VI) = 50  $\text{mg l}^{-1}$ ,  $j = 1.6 \text{Adm}^{-2}$ , Anode Surface = 50  $\text{cm}^2$ , Time of Electrolysis = 40 min



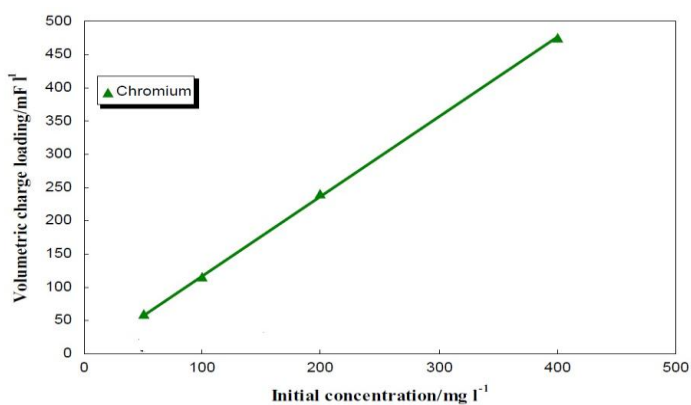
**Fig.3** Effect of Current Density on the Removal Rate of Cr(VI) Initial Concentrations of Cr(VI) = 50 mg l<sup>-1</sup>, Anode Surface = 50 cm<sup>2</sup>



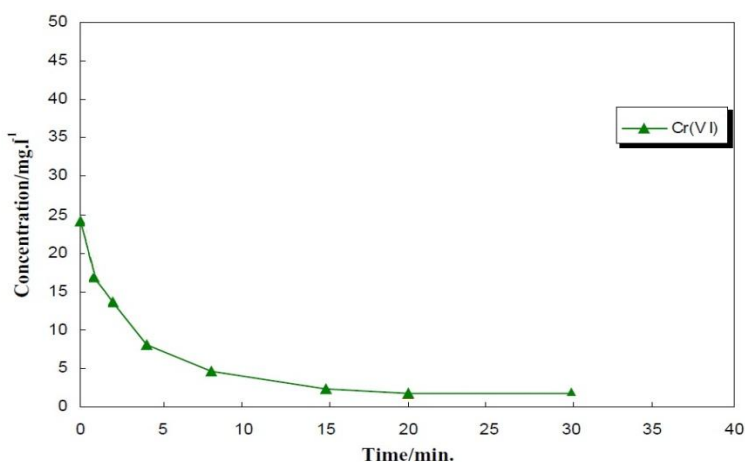
**Fig.4** The Effect of Current Density on Charge Loading. Initial Concentrations Cr(VI) = 50 mg l<sup>-1</sup>, Anode Surface = 50 cm<sup>2</sup>



**Fig.5** The Effect of Initial Concentration on the Charge Loadings Required for an Effective Removal of Cr(VI) j = 4.8Adm<sup>-2</sup>, Anode Surface = 50 cm<sup>2</sup>



**Fig.6** Effect of Electrolysis Time on the Residual Concentrations and COD Content.  $j = 4.8 \text{Adm}^{-2}$ , Anode Surface =  $50 \text{ cm}^2$



This result proves that the amount of aluminum delivered per unit of pollutants removed is not affected by the initial concentration. In addition, the charge loading required to remove chromium to the admissible level. This confirmed the less efficient removal of chromium that longer electrolysis time is necessary for chromium removal. Indeed, at high initial concentration ( $200 \text{ mg l}^{-1}$ ), 1 h was necessary to achieve the efficient removal of chromium.

### Treatment of an Industrial Wastewater

To validate the suitability of electrocoagulation for the treatment of industrial wastewater, electrolysis was carried, using a wastewater sample, collected from an electroplating unit and the residual concentrations of Cr(VI) together with COD were measured at different times of electrolysis.

The initial concentrations of Cr ( $24 \text{ mg l}^{-1}$ ) were shown to exceed the prescribed legal limits. In addition, the COD content of the wastewater ( $302 \text{ mg l}^{-1}$ ) was found to be more than two times the authorized limit, indicating the presence of some organic

compounds which were added to the electroplating bath as brighteners, levelers or wetting agents.

It appears from the results shown in Fig. 6 that the residual concentrations of chromium decreased more slowly and reached  $2 \text{ mg l}^{-1}$  after an electrolysis time of 20 min. The removal rates of metal ions seems to be relatively slow compared to the removal from synthetic wastewater. This, probably, resulted from the presence of organic compounds which may competitively adsorb on  $\text{Al}(\text{OH})_3$  coagulant, leading to a substantial reduction of metal ions removal.

The measured COD decreased from  $302 \text{ mg l}^{-1}$  to less than  $110 \text{ mg l}^{-1}$  after 25 min., which corresponds to a removal efficiency of about 64%. Beyond that time, the residual COD reached a plateau and remained nearly constant. The electrode and volumetric electricity consumptions needed to achieve an effective treatment of the studied industrial wastewater were found to be  $1 \text{ g l}^{-1}$  and  $32 \text{ A h l}^{-1}$ , respectively. All these results give an indisputable evidence that electrocoagulation can effectively reduce metal ions to a very low level. Dissolved organic compounds present in electroplating



unit wastewater are also removed. Consequently, electrocoagulation could be an efficient method for heavy metal removal from industrial wastewater.

In conclusion, the results of this study have shown the applicability of electrocoagulation in the treatment of electroplating wastewater containing chromium. The most effective removal capacity was achieved in the pH range between 4 and 8. The treatment rate was shown to increase upon increasing the current density. Whereas, 20 min. were needed to achieve an equivalent removal of Cr(VI). The slower removal of chromium attributed to a difference in the removal mechanisms. Moreover, the charge loading required to achieve an effective treatment, increased with initial concentration. In comparison to chemical coagulation, where several hours are needed and adsorption on activated carbon [Dharnaik and Ghosh, 2014], the electrocoagulation method achieves faster removal of pollutants.

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